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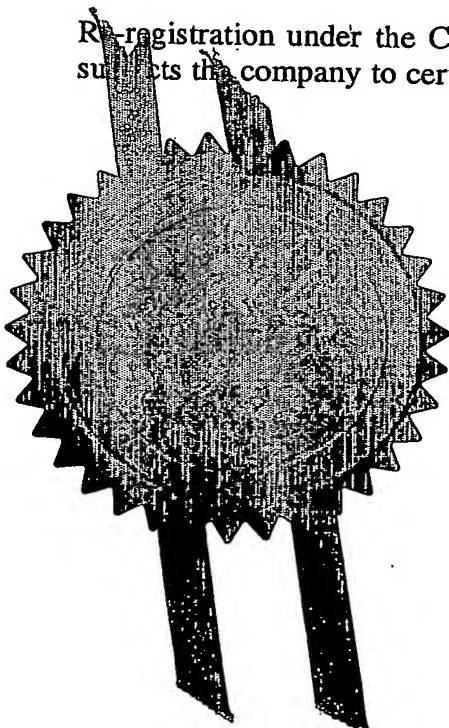
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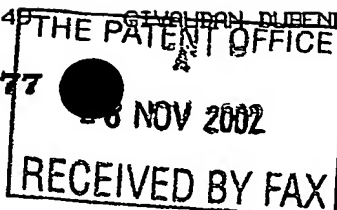
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20715 GB

28NOV02 E766793-1 D10149

2. Patent application number

(The Patent Office will fill in this part)

0227681.4

J 0.00-0227681.4

3. Full name, address and postcode of the or of each applicant *(underline all surnames)*Givaudan SA
Chemin de la Parfumerie 5
1214 Vernier
SwitzerlandPatents ADP Number *(if you know it)*

8516445001

If the applicant is a corporate body, give the country/state of its incorporation

Switzerland

4. Title of the Invention

Organic compounds

5. Name of your agent *(if you have one)*"Address for service" in the United Kingdom to which all correspondence should be sent *(including the postcode)*Centre for Innovative Technology (Givaudan UK Ltd.)
76-80 Church Street, Staines
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8447815001

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Number of earlier application

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Description 9

Claim(s) 1

Abstract 1

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DUPLICATE

Organic compounds

The invention relates to novel compounds for imparting acetaldehyde to flavor or fragrance compositions or food products or fragrance products (acetaldehyde precursors). These acetaldehyde precursors release acetaldehyde under certain conditions in said products.

It is known that acetaldehyde is a valuable ingredient in a wide variety of fresh and prepared food products, where it adds a fresh character, e.g. in vegetables such as carrot, celery, cucumber, mushroom, onion, garlic, peas, potato, and tomato, in spices such as ginger, cumin, fennel, mustard and rosemary, and in fruits, e.g. apple, arctic bramble, banana, bilberry, cherry, citrus fruits, cranberry, currants, grape, morello, olive, orange, passionfruit, peach, plum, red berry, strawberry and raspberry, but also in meat products, dairy products, bakery and confectionery products (B. Byrne and G. Sherman, "Stability of Dry Acetaldehyde Systems", Food Technology, 38(7), 57-61, 1984. The fresh character of the acetaldehyde is highly desirable for consumers especially in orange juice and other citrus juices, and therefore in the corresponding flavors and flavor compositions.

However, adding acetaldehyde to flavor or fragrance compositions or to food or fragrance products has the following drawbacks. Concentrated liquid preparations containing acetaldehyde are difficult to handle due to their high volatility and inflammability. Furthermore, acetaldehyde is chemically unstable and may polymerize, oxidize or combine with other materials or itself in presence of an acid or a base.

Flavor or fragrance preparations containing acetaldehyde have a low flash point and are not desirable for safety reasons. Since it is difficult to apply directly, spray dried acetaldehyde is a widely used product. However, due to its high volatility, significant losses during spray drying may occur. Apart from being difficult to entrap, acetaldehyde is not stable in many systems and a significant amount is rapidly lost when exposed to the atmosphere under ambient conditions, or even in packaged form, if small amounts of water are present.

The intractability of acetaldehyde has been addressed in various approaches, including the protection of the molecule in a matrix containing starches and sugars, which can be done by spray drying (B. Byrne and G. Sherman, "Stability of Dry Acetaldehyde Systems", Food Technology, 38(7), 57-61, 1984.). However, the spray drying system has been shown to be not as effective as a system employing

chemicals that degrade to release acetaldehyde (R. S. DeSimone and B. Byrne, "Aldehyde Generator for Flavors", *Perfumer & Flavorist*, Vol. 11, p. 15-26, 1987).

US 4, 280, 011 and US 5, 079, 023 disclose several chemical precursors which are acetals that hydrolyze to give acetaldehyde. One of these chemical precursors is 1, 2-di[(1'-ethoxy)ethoxy]propane (ALDEMAX, CAS 67715-79-1). A limitation to the usefulness of acetals which release their corresponding aldehydes is that they provide their own characteristic aroma (flavor or odor), which often might not be desired.

US 4,781,762 discloses another type of chemical precursor of acetaldehyde that incorporates the functionality of a carboxylate. These precursors release acetaldehyde and have relatively short half-lives in the order of minutes in acid media.

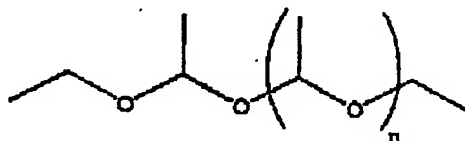
However, the known acetaldehyde precursors are prone to decompose during spray-drying or the compounding of flavors or fragrances, or when exposed to water or even atmospheric humidity. Compounding with other ingredients such as alcohols and acids might lead to reactions of the acetaldehyde precursors to yield undesired by-products, e.g. various hemi-acetals and acetals. Together with propylene glycol, which is used frequently in the flavor industry, stable acetals are formed that are perceived organoleptically as off-flavors. Even relatively stable precursors e.g. acetals such as 1, 2-di[(1'-ethoxy)ethoxy]propane may hydrolyse prematurely under unfavorable conditions occurring during flavor or fragrance compounding, formulation into a food or fragrance product, or during transport and storage.

Another limiting factor to the effectiveness of chemical precursors relates to their release time. Known precursors do not release acetaldehyde fast enough in some applications depending on factors such as the pH of the release media. A further factor relates to regulatory requirements: These compounds are classified as "artificial" since their structures do not occur in nature. Flavors made with known acetaldehyde precursors cannot be designated as natural flavors.

There remains a need for chemical precursors which are chemically inert and stable under formulation, transport and storage conditions, but not so that they do not release acetaldehyde rapidly when required. Furthermore, they should not result in undesired side products and should not interfere with the aroma or taste of the desired flavor or fragrance.

Surprisingly, the problems associated with the prior art are overcome with the provision of new acetaldehyde precursor compounds.

Therefore, the invention provides in a first aspect an acetaldehyde precursor according to formula (I) with $n = 1, 2, 3, 4$ or larger.



formula (I)

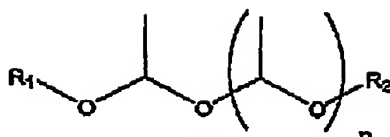
The acetaldehyde precursor of formula (I) with $n=1$ is stable under neutral pH conditions but rapidly releases acetaldehyde under acidic conditions. The half life times of an acetaldehyde precursor of formula (I) with $n=1$ at pH 2.8, 3.18, 3.81 and 4.14 are calculated from the measurements described in the following description and indicated in the table below.

pH value	2.8	3.18	3.81	4.14
rate constant k in 1/second*	0.005966	0.003446	0.00069	0.000382
half life time λ in seconds	116	201	1004	1813

* Precursor + $H_2O \rightarrow$ Ethanol + Acetaldehyde

The acetaldehyde precursor of formula (I) with $n=1$ releases Acetaldehyde significantly faster (Half-life pH 3.0 = 306 seconds, pH 3.5 = 966 sec.) than 1, 2-di[(1'-ethoxy)ethoxy]propane (Half-life at pH 3.0 = 625 sec, pH 3.5 = 3906 seconds) and faster than the monomer, acetaldehyde diethyl acetal, (Half-life at pH 3.0 = 579 sec, pH 3.5 = 2493 sec.). Since the acetaldehyde precursor according to formula I with $n=1$ (dimer) releases acetaldehyde faster than the monomer with $n=0$, the trimer with $n=2$ is expected to release acetaldehyde at least as rapidly as the dimer.

As will be apparent to the skilled person, alternative compounds of formula II wherein R1 and R2 may be selected from linear, branched or cyclic alkyl, aryl, alcohols or sugar residues, and n is larger than 4, for example 5, 6, 7, 8, 9, 10, may also act as acetaldehyde precursors. As will be apparent to the skilled person, compounds of formula (II) with increasing n value, decrease in aqueous solubility, and therefore R1 and R2 should be selected accordingly from hydrophilic residues such as alcohols or sugars, so that the compound is still soluble.



formula (II)

The release rate of acetaldehyde from the precursor according to formula I with $n=1$ is measured and compared to 1, 2-di[(1'-ethoxy)ethoxy]propane and acetaldehyde diethyl. The measurements are conducted as described in S. M. Oon and D. G. Kubler, "Hydrolysis of Aldal Acetals", *J. Org. Chem.*, 1982, 47, 1166-1171. A phosphate buffer solution (2 mL) is placed into a quartz cuvette and 5 mg of the respective acetaldehyde precursor is added. The mixture is mixed well and sealed using Parafilm. The release of acetaldehyde is monitored in a UV-Photometer measuring the increase of the extinction at 280 nm over time. Depending on reaction speed, all 3 to 10 seconds measurements are conducted. Obtained formation curves of acetaldehyde at pH 2.8, pH 3.18, pH 3.81 and pH 4.14 are very well represented by the formula $C_t = C_0 + C_x \times (1 - e^{-kt})$, wherein C_t the absorption corresponding to the concentration of acetaldehyde at time t , C_0 absorption corresponding to the concentration of acetaldehyde at time $t = 0$, C_x the total amount of acetaldehyde present in the precursor, k the rate constant and t the reaction time. The rate constants are calculated by a numeric approximation program (TABLE CURVE 2D, © AISN Software Inc.). The half life time λ is calculated from the rate constant using the equation $\lambda = -\ln(0,5)/k$.

Application of acetaldehyde precursors of the present invention in food and fragrance products or formulations dramatically reduces the risk of ignition and explosion: whereas the flash point of an orange flavor A (example 1) with 10% acetaldehyde is below -10°C , the flash point of an orange flavor B (example 1) with an acetaldehyde precursor corresponding to 10% acetaldehyde according to formula I with $n=1$ is $+48^\circ\text{C}$.

An acetaldehyde precursors according to formula (I) with $n=1$ occurs in nature and therefore does not have to be designated as "artificial" in food products for regulatory reasons. A sample of REDD NATURAL ACETALDEHYDE FRAC (Givaudan Lakeland, Florida) is analysed by GC-MS using 20715 GB

splitless injection mode on a DB-1 and DB-WAX column. The mass spectrum and retention time matches the reference substance 1-ethoxy-1-(1-ethoxy-ethoxy)-ethane (compound according to formula (I) with $n=1$, CAS 80243-06-7). The retention indices based on methyl ester references are 631 on a DB-1 (J&W, Palo Alto, USA, 30 m x 0.32 mm i.D. x 0.25 μ m film, Part 123-1032) column and 578 on a DB-WAX column (J&W, Palo Alto, USA, 30 m x 0.32 mm i.D. x 0.25 μ m film thickness, Part 123-7032).

Apart from a rapid release of acetaldehyde under acidic conditions a further advantage of acetaldehyde precursors according to the present invention is that they have a less pronounced aroma compared to acetaldehyde, diethyl acetal or 1, 2-di[(1'-ethoxy)ethoxy]propane.

In a second aspect, the present invention provides flavors or fragrances, preparations of flavors or fragrances, and food or consumer products containing flavors or fragrances or preparations of flavors or fragrances, comprising acetaldehyde precursors of formula I with $n=1$, $n=2$, or $n=3$, or mixtures thereof.

Acetaldehyde precursors according to the present invention may be formulated in various forms well known to a person skilled in the art, including solid, spray dried or encapsulated e.g. granulated, extruded, coacervated, liquid or as an emulsion.

Acetaldehyde precursors according to the present invention may be employed in flavor or fragrance formulations, or directly in food or fragrance products, e.g. in cereal products, rice products, tapioca products, sago products, baker's products, biscuit products, pastry products, bread products, confectionery products, desert products, gums, chewing gums, chocolates, ices, honey products, treacle products, yeast products, baking-powder, salt and spice products, savory products, mustard products, vinegar products, sauces (condiments), tobacco products, cigars, cigarettes, processed foods, cooked fruits and vegetable products, meat and meat products, jellies, jams, fruit sauces, egg products, milk and dairy products, cheese products, butter and butter substitute products, milk substitute products, soy products, edible oils and fat products, medicaments, beverages, alcoholic drinks, beers, soft drinks, mineral and aerated waters and other non-alcoholic drinks, fruit drinks, fruit juices, coffee, artificial coffee, tea, cocoa, or in food products such as including forms requiring reconstitution, food extracts, plant extracts, meat extracts, condiments, sweeteners, nutraceuticals, gelatins, pharmaceutical and non-pharmaceutical gums, tablets, lozenges, drops, emulsions, elixirs, syrups and other preparations for making beverages, instant beverages and effervescent tablets, and combinations thereof.

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Especially preferred applications include beverages such as soft drinks, mineral and aerated waters and other non-alcoholic drinks, fruit drinks, fruit juices, and preparations for making beverages including tablets, syrups, instant beverages, effervescent tablets.

The amount of the acetaldehyde precursor added to the foodstuff or fragrance is sufficient to impart the desired fresh flavor, aroma or fragrance. The person skilled in the art will appreciate that the precise amount will vary in wide ranges according to the foodstuff/fragrance or flavor/fragrance composition to be flavored/fragranced and the flavor/fragrance impact sought.

Generally, 0.1 to 500 mg/kg, preferably 0.1 – 250 mg/kg, more preferably 0.1-100 mg/kg in a food or a beverage may be an organoleptically useful range. The person skilled in the art acknowledges that depending on the application the actual concentration will be within or without of this range in products or formulations.

Acetaldehyde precursors according to the present invention may be present in flavor or fragrance formulations in a concentration of 0.01 – 1000 g/kg, preferably 0.01 – 500 g/kg more preferably 0.1 – 200 g/kg.

A person skilled in the art will appreciate that formulations and products may contain additional ingredients which may comprise various additives and excipients well known in the art, including anti-caking agents, anti-foaming agents, anti-oxidants, binders, colorants, diluents, disintegrants, emulsifiers, encapsulating agents, enzymes, fats, flavor-enhancers, flavoring agents, gums, lubricants, polysaccharides, preservatives, proteins, solubilisers, solvents, stabilisers, sugar-derivatives, surfactants, sweetening agents, vitamins, waxes, and the like. Solvents which may be used are known to those skilled in the art and include e.g. ethanol, ethylene glycol, propylene glycol, glycerin, triacetin, diethyl phthalate and dimethyl phthalate. Encapsulants and gums include maltodextrin, gum arabic, alginates, gelatin, modified starch, and polysaccharides. Examples of additives, excipients, carriers, diluents or solvents for flavor or fragrance compounds may be found e.g. in „Perfume and Flavor Materials of Natural Origin“, S. Arctander, Ed., Elizabeth, N.J., 1960; in "Perfume and Flavor Chemicals", S. Arctander, Ed., Vol. I & II, Alured Publishing Corporation, Carol Stream, USA, 1994; in "Flavourings", E. Ziegler and H. Ziegler (ed.), Wiley-VCH Weinheim, 1998, and "CTFA Cosmetic Ingredient Handbook", J.M. Nikitakis (ed.), 1st ed., The Cosmetic, Toiletry and Fragrance Association, Inc., Washington, 1988.

Acetaldehyde precursors according to the present invention are suitable for applications in products with acidic pH e.g. soft drinks or instant beverages and effervescent tablets. The pH in the product ready made drink may be from 2 to 4.5, preferably 2.5 to 4, more preferably from about 2.6 to 3.0.

However, the respective amount of acetaldehyde precursor employed will depend on the respective application, e.g. the pH and desired release rate and desired flavor or fragrance intensity. It will be apparent to the person skilled in the art that for specific applications an amount within or outside of this range may be employed.

In another aspect, the present invention provides the use of acetaldehyde precursors according to formula (I) with $n=1, 2$ or 3 , or mixtures thereof in preparations of flavors or fragrances, food or consumer products containing flavors or fragrances or preparations of flavors or fragrances.

The synthesis of 1-ethoxy-1-(1-ethoxy-ethoxy)-ethane may be carried out by modifying a procedure described by Oon and Kubler (S. M. Oon and D. G. Kubler, "Hydrolysis of Aldal Acetals", *J. Org. Chem.*, 1982, 47, 1166-1171), as is further set out in the Examples. Furthermore, applicant has found that molecules according to formula (I) with n larger than 1, e.g. 2, 3, 4 or larger can be produced analogously to the aforementioned synthesis by employing acetaldehyde increasingly larger excess.

Alternatively, compounds according to formula I may be prepared from acetaldehyde and ethanol using a molecular sieve, for example, a 6A acidic molecular sieve (CBV8014) may be used.

A mixture of compounds according to formula 1 with $n=1$ and $n=2$ may be synthesized by reacting acetaldehyde and ethanol in hexane a catalyst, for example tartaric acid, and if desired, compounds with particular n values may be isolated from the mixture using separation techniques known in the art.

Samples prepared to above-mentioned processes may significantly increase the yield of acetaldehyde during spraydrying and minimize losses during storage. 1-ethoxy-1-(1-ethoxy-ethoxy)-ethane itself can be used as nature identical acetaldehyde precursor in dry flavour formulations.

Examples

Example 1: Synthesis of 1-ethoxy-1-(1-ethoxy-ethoxy)-ethane (compound of Formula (I) where $n=1$)

In a 500 mL round bottom flask anhydrous hydrogen chloride gas (91 g, 2.5 mol, Aldrich) was bubbled into acetaldehyde (100 g, 2.25 mol, Aldrich) which was cooled to -10°C to -20°C . After the addition of hydrogen chloride gas was complete the mixture was cooled to -40°C to crystallize out the newly formed water. The solution was filtered, dried over CaCl_2 , filtered again, and distilled to give the intermediate, dichlorodiethyl ether (68.68 g, 0.48 mol, 42.7 % yield). This material was not further characterized and was used immediately in the next step.

Under nitrogen gas in a 500 mL round bottom flask dichlorodiethyl ether (68.68 g, 0.48 mol) was added dropwise to a sodium ethoxide solution prepared from ethyl alcohol (360 mL) and sodium (23.6 g, 1 mol) cooled to -10°C to -20°C . The reaction was stirred and allowed to warm up to room temperature. The solution was filtered to remove salt and distilled (24 mmHg, $65-75^{\circ}\text{C}$) to give 1-ethoxy-1-(1-ethoxy-ethoxy)-ethane (10.5 g, 0.06 mol, 12 % yield). ^1H NMR ref: 4.85 q, 3.50 dq, 1.23 d, 1.11 t; ^{13}C NMR: 98.86, 60.745, 21.12, 15.70; MS: 45, 73, 89, 103, 117, 147

Example 2: Acetaldehyde precursors in Orange Flavour Reduces Flash Point.

Orange flavour A with 10% acetaldehyde (w/w) shows a flash point of below 4°C . Orange flavour B, where acetaldehyde was substituted by an acetaldehyde precursor corresponding to 10% acetaldehyde according to formula I with $n=1$ the flash point is $+48^{\circ}\text{C}$.

	Orange flavour A with acetaldehyde	Orange flavour B with acetaldehyde precursor
orange oil (Brazil) single fold	77.5 g	77.5 g
orange oil (Brazil) 5-fold	5 g	5 g
orange oil (Brazil) 25-fold	5 g	5 g
ethyl butyrate (CAS 105-54-4)	2.5 g	2.5 g
acetaldehyde (CAS 75-07-0)	10 g	-
compound acc. to formula I with $n=1$	-	18.4 g

Example 3: Acetaldehyde precursors in spray dried aroma for instant beverage.

The orange aromas A and B from Example B were spraydried after addition of maltodextrin, and water. The resulting powders were mixed with an instant beverage base and stored at room temperature for 14

days. The instant beverages were dissolved in water (160g/L). The instant beverage B was more juicy, fresh and reminiscent on fresh squeezed orange juice compared to instant beverage A.

Spray dried formula

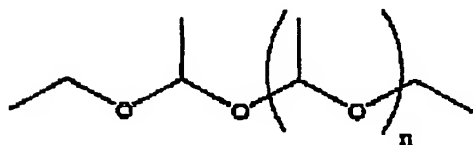
	<u>Spraydried Aroma A</u>	<u>Spraydried Aroma B</u>
Orange flavour A	110	-
Orange flavour B	-	110
Water	445	445
Maltodextrine (CAS 9050-36-6)	400	400
Starch octenyl succinate (CAS 9087-61-0)	45	45

Instant Beverage formula

	<u>Instant beverage A</u>	<u>Instant beverage B</u>
Spraydried aroma A	1.2	-
Spraydried aroma B	-	1.2
Citric acid (CAS 77-92-9)	61.5	61.5
Saccharose (CAS 57-50-1)	926.5	926.5
Titanoxide (CAS 13463-67-7)	3.8	3.8
Pectin (CAS 9000-69-5)	3.8	3.8
Trisodium citrate (CAS 77-92-9)	3.2	3.2

Claims

1. An acetaldehyde precursor according to formula (I) with $n = 1, n=2, n=3$ or $n=4$.

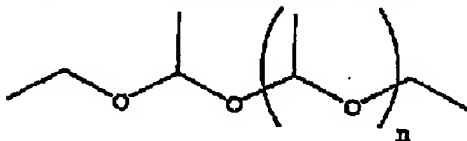


formula (I)

2. Flavors or fragrances, preparations of flavors or fragrances, and food or consumer products containing flavors or fragrances or preparations of flavors or fragrances, comprising compounds according to formula I with $n = 1, n=2, n=3$, or $n=4$, or mixtures thereof.
3. Flavors or fragrances, preparations of flavors or fragrances, and food or consumer products containing flavors or fragrances or preparations of flavors or fragrances according to claim 2, in an amount sufficient to provide a fresh type of flavor or fragrance.
4. Spray-dried composition comprising compounds or compositions according to any of the previous claims.
5. Use of compounds according to formula I with $n = 1, n=2, n=3$, or $n=4$, or mixtures thereof, in flavors or fragrances, in preparations of flavors or fragrances, and in food or consumer products containing flavors or fragrances or preparations of flavors or fragrances.

Abstract

The invention relates to novel compounds for imparting acetaldehyde to flavor or fragrance compositions or food products or fragrance products (acetaldehyde precursors). These acetaldehyde precursors release acetaldehyde under certain conditions in said products. Acetaldehyde precursors are compounds according to formula (I)



formula (I)

PCT Application

CH0300784



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